# SYNTHESIS OF GRAPITE-SIC MICRO-COMPOSITE AND ITS EFFECT ON THE MECHANICAL STRENGTH OF TEMPERED MAGNESIA-CARBON COMPACTS.

A THESIS SUBMITTED IN PARTIAL FULFILLMENT

OF THE REQUIREMENTS FOR THE DEGREE OF

BACHELOR OF TECHNOLOGY

IN

CERAMIC ENGINEERING


SUBMITTED BY

SAIMON RAY

(ROLL NO- 109CR0661)


Under the guidance of

PROF. DEBASISH SARKAR



Department of Ceramic Engineering

National Institute of Technology

Rourkela, Odisha-769008


# *CERTIFICATE*

This is to certify that the thesis entitled, "**Synthesis of grapite-sic micro-composite and its effect on the mechanical strength of tempered magnesia-carbon compacts**" submitted by **SAIMON RAY** in partial fulfillment of the requirement for the award of **Bachelor of Technology** Degree in **Ceramic Engineering** at **National Institute of Technology, Rourkela** is an authentic work carried out by him under my supervision and guidance.

To the best of my knowledge, the matter embodied in the thesis has not been submitted to any other University/ Institute for the award of any Degree or Diploma.

Date:

PROF. DEBASISH SARKAR
Dept. of Ceramic Engineering
National Institute of Technology
R0urkela-769008

# ACKNOWLEDGEMENT

I acknowledge and I am thoroughly indebted to all those who have been instrumental in helping me out with the various facets of my project.

With deep regard and profound respect, I would like to express my gratitude to Prof. H. S Maiti, INAE Distinguished Professor, Department of Ceramic engineering, NIT ROURKELA for playing a pivotal role in steering the project into a reality. At the same time, I extend a note of sincere and benevolent thanks to my guide PROF. DEBASISH SARKAR without whose guidance and constant support this project would have been a mere impossibility.

I am also thankful to all the faculty members of the Department of Ceramic Engineering NIT Rourkela for their all-out support.

It is difficult to record my deep sense of appreciation for the technical support received from a large number of M. Tech and Ph. D scholars of the department individually. However, I shall be failing in my duty if I do not mention the extensive support received particularly from Mr. Ganesh Sahoo, Mr. Sanjay Swain and Mr. Raju Mula throughout the entire duration of this project.

Last but not the least I thank almighty, my parents and near and dear ones for being a strong pillar of emotional and financial support during this endeavor of mine.

*A HUMBLE SUBMISSION BEFORE YOU ALL* !!!!

*Saimon Ray*

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# ABSTRACT

The present study relates to the field of ceramic powders. Particularly, it comprises a process for producing the graphite - silicon carbide (SiC) micro-composite ceramic powders by carbo-thermal reduction.

Silicon carbide is a ceramic material valued mainly for its high resistance to thermal stress and shock and its exceptional corrosion resistance in high temperature oxidizing environments. In this project work, in particular, an attempt has been made to synthesize a graphite-SiC micro-composite by carbo-thermal reduction process and its effects on mechanical properties of magnesia-carbon compacts. The graphite – SiC composite has been synthesized by in-situ formation of SiC on graphite through carbo-thermal reduction of graphite and microfine silica. Attempts have also been made to optimize different parameters for synthesis of the composites and the variation in mechanical properties of magnesia-carbon compacts with the addition of these composites.

# CHAPTER-1

# INSIGHT

# INTRODUCTION

Synthesis of nonoxide composite powder is the process of blending fine powdered materials of specific proportions and then heating the blended material in a controlled atmosphere to carry out chemical reactions between the powders at elevated temperature. Silicon carbide is an important non-oxide ceramic which has diverse industrial applications. It can be used in oxidizing temperature up to 1350$^{o}$C a useful material in various acid, caustic, corrosive, abrasive, or high temperature environments [1 - 3]. A number of methods of manufacturing process of silicon carbide have been developed. The most widely accepted "Acheson process" is used for large-scale manufacturing. Many other methods of manufacturing silicon carbide are (1) the carbo-thermal reduction of silica and carbon in an inert atmosphere within a vertical furnace; (2) a direct reaction of silicon powder and fine carbon powder at around 1400 $^{o}$C temperature with inert atmosphere; (3) a sol-gel silica/carbon reduction process; and (4) a two-stage synthetic silica/carbon reduction process, which is also carried out as a gas phase reaction.

Generally, silicon carbide exists in both an alpha and a beta form. The alpha phase is categorized as hexagonal, but exhibits poly-types based upon stacking sequences in the layered structure. The beta phase is in cubic form. In both of these structures, each atom is tetrahedrally surrounded by four atoms of the other species, forming strong covalent bonds. Alpha silicon carbide is assumed to be the stable at high temperature compare cubic – beta silicon carbide.

In our present work, an attempt has been made to produce graphite-SiC micro-composite by carbo-thermal process, which has a stable β-phase of SiC. From experiments, we found that this composite has two types morphology of SiC formed. One is irregular worm like ribbon structure, another is fiber or whisker type morphology. Our aim is to improve the mechanical properties of MgO-C compacts by replacing a part of the graphite by these micro composites. SiC has higher mechanical strength, hot strength, oxidation resistance than graphite. At the same time it is a less wet-able material than the graphite particularly with respect to softened pitch which is considered as a binder.

# CHAPTER -2

# LITERATURE REVIEW

## 2.1 Silicon Carbide:

Silicon carbide was discovered by a Swedish scientist Berzelius in 1824. Silicon carbide can occur in more than 230 crystalline forms called polytypes. The most common ones are: 3C, 4H, 6H and 15R. Silicon carbide has excellent chemical, mechanical and thermal properties which made them applicable in structural, high temperature, and electronics applications. The simplest manufacturing process of SiC is to combine silica sand and carbon in an "Acheson graphite electric resistance furnace" at temperatures higher than 2500°C [1]

### 2.1.2 Structure of silicon carbide:

SiC has more than 230 different polymorphs called polytypes. The different surface energies of Silicon and Carbon faces have influenced the formation of different polytypes nuclei [1, 4]. The most common polytypes include 3C, 2H, 4H, 6H, 8H, 9R, 10H, 14H, 15R,19R, 2OH, 21H, and 24R, where (C), (H) and (R) are the three basic cubic, hexagonal and rhombohedral crystallographic categories. In the cubic zinc-blende structure, characterized as 3C-SiC or β-SiC, Si and C occupy ordered sites in a diamond framework. SiC polytypes are discriminated by the stacking sequence of each tetrahedrally bonded Si-C bilayer. In fact the individual polytypes differ in both band gap energies and electronic properties. So the band gap differs with the polytypes; 2.3 eV for 3C-SiC, 3.0 eV for 6H-SiC and 3.2 eV for 4H-SiC. As a result of its smaller band gap, 3C-SiC has many advantages compared to the other polytypes, which allows inversion at lower electric field strength. Alpha silicon carbide (α-SiC) is the most commonly encountered polymorph; it is the stable form at elevated temperature as high as 1700°C and has a hexagonal crystal structure (similar to Wurtzite). Among all

the hexagonal structures, 6H-SiC and 4H-SiC are the only SiC polytypes presently available in bulk wafer form. The β-SiC (3C-SiC) with a zinc blend crystal structure (similar to diamond), is formed at temperatures below 1700°C .The number 3 refers to the number of layers required for periodicity. 3C-SiC possesses the smallest band gap (~2.4eV).

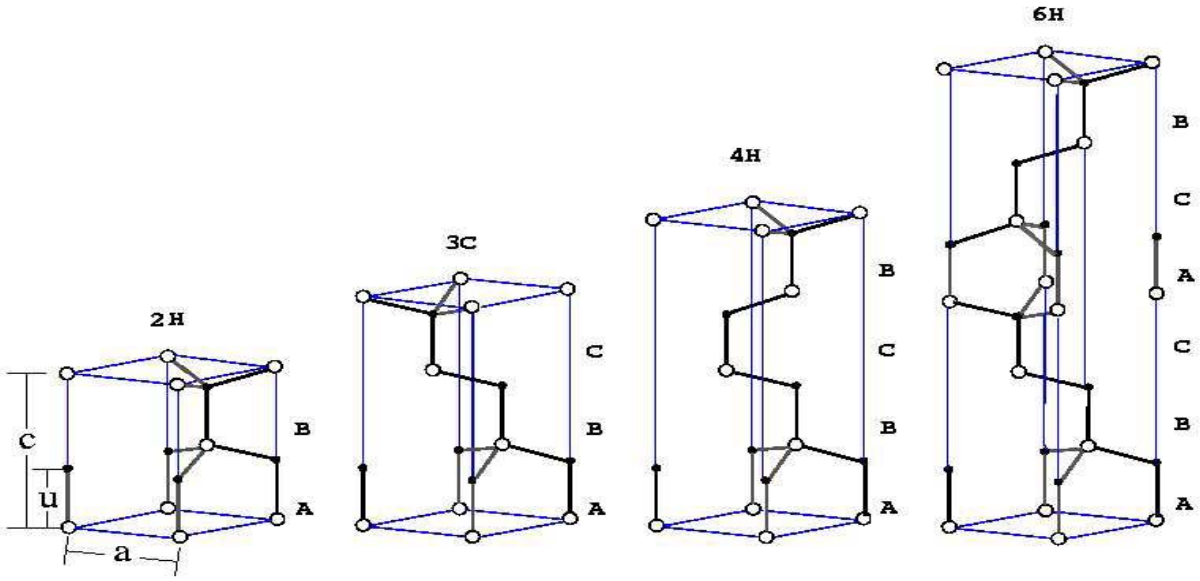



**Fig 2.1**. Schematic representation of the staking sequence of hexagonal SiC bilayers for 2H, 3C, 4H and 6H polytypes [4]

### 2.1.3 Synthesis of SiC:

### 2.1.3.1 Acheson Process:

Naturally silicon carbide is found only in very little quantities in certain types of meteorites [1]. The most encountered SiC materials are thus synthetic. Traditionally, SiC materials have been produced through the Acheson process, in which an Acheson graphite electric resistance furnace, which is used for production of poly-crystalline SiC that is suitable for grinding and cutting applications. In this process a solid-state reaction between silica sand and petroleum coke at very high temperature (more than 2500°C) leads to the formation of silicon carbide under the general reaction 2.1

$$SiO_2(s) + 3C(s) \rightarrow SiC(s) + 2CO\ (g) \quad \ldots\ldots (2.1)$$

Crystalline SiC achieved by the Acheson-Process happens in different polytypes and differs in purity.

### 2.1.3.2 Synthesis of SiC by Chemical Vapor Deposition (CVD):

CVD is one of the most suitable used methods to produce SiC in various shapes of thin films powders, whiskers and nano-rods using Si-C-HCl system. Amorphous fine silicon carbide powder has been prepared by CVD method in the $SiH_4$-$C_2H_2$ system under nitrogen as a carrying gas. Reaction is expected to take place in the reaction zone as follows;

$$2\ SiH_4(g) + C_2H_2(g) \rightarrow 2SiC\ (g) + 5H_2(g) \quad \ldots\ldots\ldots (2.2)$$

The reported particle size of SiC prepared in this process varies in the range 0.1- 0.5μm [1, 2].

### 2.1.3.3 Sol-gel processing technique for synthesizing SiC:

The sol-gel process involves two main steps that are hydrolysis and poly-condensation [5]. The first one starts with the preparation of a Silica-glass by blending an appropriate alkoxide as precursor, with water and a common solvent to form a solution. Hydrolysis leads to the formation of silanol groups (SiOH) consequently condensed to produce siloxane bonds (SiOSi). After solvent removal, it needs a drying process; a xerogel is formed, accompanied by a significant shrinkage and densification. This phase of processing affects profoundly the ultimate microstructure of the final component. However, for industrial applications, this process is relatively expensive, as it requires costly precursors, particularly compared to the Acheson process [1, 5].

### 2.1.3.4 Synthesis of SiC fibers and whiskers:

SiC fiber is considered to be more suitable reinforcement material for the composites. SiC fibers have much higher aspect ratio compared to SiC whiskers. For synthesis of nano-fiber a number of approach has been suggested such as chemical vapor deposition (CVD), carbon nanotube confined reaction, sol-gel route, carbo-thermal reduction, arc discharge etc. [1]. During the formation of SiC nano fibers through carbo-thermal reduction several intermediate reaction steps occur. In the initial stage solid-solid reaction (2.3) occurs, then a solid-gas phase reaction (2.4) and gas phase (2.5) reaction occurs.

$$SiO2(S) + C(S) \rightarrow SiO(g) + CO(g) \quad \ldots\ldots\ldots \quad (2.3)$$

$$SiO(g) + 2C(S) \rightarrow SiC(S) + CO(g) \quad \ldots\ldots\ldots \quad (2.4)$$

$$SiO(g) + 3CO(g) \rightarrow SiC(S) + 2CO_2(g) \quad \ldots\ldots\ldots \quad (2.5)$$

## 2.2 Raw Materials for MgO-C compacts:

### 2.2.1 Magnesia:

Magnesia is known as a basic refractory material. It is the main constituent of MgO-C brick [6, 7]. Generally, there are three different kinds of magnesia raw materials are used to produce MgO-C bricks (a) sea water magnesia (b) dead burn magnesia (c) fused magnesia. Sea-water magnesia has average particle size around 300μm, dead burn magnesia has around 500μm, fused magnesia has around 1000μm. Quality of magnesia grain has the most significant effect on the corrosion resistance. In this project work MgO-C compacts has been prepared by fused magnesia.

### 2.2.2 Graphite:

Natural flake graphite with high purity and high crystalinity is used in MgO-C refractory. Generally, graphite is used for its non-wetting characteristics, which provides the maximum corrosion resistance from the corrosive slag, molten metal and minimizes the slag penetration. The particle size of the graphite has major effects on modulus of elasticity and permeability. The bigger graphite particle size reduces the strength of MgO-C bricks particularly during heat treatment or processing of steel [8]. Purity of graphite should be more than 92% in order to achieve the desirable property of corrosion resistance.

### 2.2.3 Pitch

Binder Pitch is resulting from the processing of high temperature coal tar. It is a black solid material at room temperature and consists of a complex mixture of predominantly aromatic hydrocarbons [9, 10]. It exhibits an extensive softening range rather than a defined melting temperature. The main source of pitch is coal tar and petroleum. The major application area of pitch is industrial electrode production, but preparation of carbon fiber, graphite and composite is also possible. The process of converting a pitch material to graphite & other high performance carbon comprises an intermediate phase called meso-phase. Pitches & meso-phase like polymers are the thermo-plastic systems. During processing, binder pitch becomes highly thermo plastic to form a homogeneous mixture with the filler.

# CHAPTER – 3

# EXPERIMENTATION

## 3.1 Synthesis of graphite-SiC micro composite:

The graphite – SiC micro-composite was prepared from graphite flake and micro-fine silica following a procedure described elsewhere [11]. In brief, graphite flake of around 212μ and micro-fine silica were used for carbo-thermal reduction within the temperature range of 1400 – 1650$^{o}$C for the time variation of 2 – 8hr. Silica content was varied within the range of 20 – 70wt%. Two distinctly different morphologies of SiC were formed on graphite during the carbo-thermal reduction; fibers were formed preferably around the edge of graphite flake and ribbon on the graphite surface. Detail microstructure and phase analysis are discussed in results and discussion section.

## 3.2 Fabrication of MgO-C Compacts:

The raw material used for MgO-C compacts were fused magnesia (FM97LC) having crystallite size in the range 800-1000μm, natural flake graphite (94FC), solid pitch as the binder.

### 3.2.1 Batch Preparation:

Different batches of MgO-C compacts have been prepared by varying the weight percentage of raw materials, binder percentage and graphite-SiC micro composites. Compositions of different batches are listed in the following tables. Solid pitch was used as the binder.

**Table 3.1**: Different batches of MgO-C with varying percentages of binder.

| Materials | Sample 1 | Sample 2 | Sample 3 | Sample 4 |
|---|---|---|---|---|
| MgO (0-710μ) | 60% | 60% | 60% | 60% |
| MgO BMF | 30% | 30% | 30% | 30% |
| GRAPHITE (94FC) | 10% | 10% | 10% | 10% |
| BINDER (Solid Pitch) % of Mix | 5% | 10% | 15% | 20% |

**Table 3.2:** Variation of graphite content in different samples.

| Materials | Sample 1 | Sample 2 | Sample 3 |
|---|---|---|---|
| MgO (0-710μ) | 60% | 60% | 60% |
| MgO BMF | 30% | 30% | 30% |
| GRAPHITE (94FC) | 10% | 15% | 20% |
| BINDER (Solid Pitch) % of Mix | 10% | 10% | 10% |

**Table 3.3** Variation of composite (both ribbon & fiber) content in different samples.

| Materials | Sample 1 | Sample 2 | Sample 3 | Sample 4 | Sample 5 |
|---|---|---|---|---|---|
| MgO(0-710μ) | 60% | 60% | 60% | 60% | 60% |
| BMF | 30% | 30% | 30% | 30% | 30% |
| Graphite + Composite | 9.5% + 0.5% | 9% + 1 % | 8.5% + 1.5% | 8% + 2 % | 7.5% + 2.5% |
| Binder | 15% | 15% | 15% | 15% | 15% |

### 3.2.2 Mixing and Aging

In case of no presence of composites, all the batches are mixed together in an agate mortar. But when, the composites are used initially they are properly blended with graphite in an agate mortar separately. Then other materials are added to it and properly mixed. After initial stage mixing, the mixed batch is heated and hot mixing is done within a temperature range 120°C-130°C for 20 minutes. Then the hot mixed batch is left for 10 minutes, and then heated & mixed for second time for 10 minutes.

### 3.2.3 Pressing:

The hot-mixed batches were pressed with a load of around 15000 Kg. in a uniaxial press to form slabs of dimensions 75mm x 20mm x 10mm. Equal amounts of mix was used to ensure identical size of the samples. After pressing, de-molding is carefully done to take out the green sample.

### 3.2.4 Tempering:

Tempering of the pressed green sample was done at 280°C for 10-12 hours. In this process volatile materials were removed from the samples and the green strength was increased by the chemical bond between the material grains and the bonding phase. As the temperature increases there is a polymorphic change in the binder pitch, which holds the whole structure and gives green strength to the pressed sample.

### 3.3.1 Characterization and measurement:

#### 3.3.1.1 Phase analysis of the micro-composite:

X-ray powder diffraction (XRD) is a fast analytical technique primarily used for phase identification of a crystalline material and can provide information on unit cell dimensions. The analyzed materials are finely ground, homogenized, and average bulk composition is determined. Phase analysis of all the samples was carried out by using Cu - Kα radiation. Different XRD patterns were examined by X'pert high score software.

#### 3.3.1.2 Micro-structural analysis of composite by SEM:

The scanning electron microscope (SEM) is used to focus a beam of high-energy electrons to generate a variety of signals at the surface of solid specimens. The SEM is also capable of performing analyses of selected point localities on the sample; this method is specifically useful in qualitatively or semi-quantitatively determining chemical compositions (using EDS), crystalline structure, and crystal orientations (using EBSD). The powders were fixed on a self-adhesive carbon tape and analysis of different region was carried out.

#### 3.3.1.3 Cold modulus of rupture (CMOR):

The modulus of rupture of refractory materials are measured by the calculating the three point bending strength of bar at room temperature. The following formula is used to calculate CMOR.

$$CMOR = (3 \times P \times L) / (2 \times b \times d^2)$$

Where "P" is the maximum load required to break the sample, "L" is the span length, "b" & "d" are the breadth and height of the sample, respectively.

# CHAPTER- 4

# RESULTS AND DISCUSSION

## 4.1 Identification of silicon carbide by XRD & SEM:

During the experiments, graphite flakes was made to react with silica at a temperature range around 1550-1650°C for 4 hours in a reducing atmosphere. After preparation of composite powders, XRD analysis of reacted powder was done, which confirmed the formation of β-SiC in the sample together with the graphite. XRD analysis reveals the presence of unreacted free silica when initial silica addition with graphite is more.

Two different SiC morphologies are confirmed from SEM images. One is beautiful silicon carbide fibers, which are grown along the edges of the original graphite flakes and also between the inter-layer spacing of the flakes generally the temperature range below 1600°C. These fibers are grown at the edges of graphite flakes but not on the flat surface of the graphite, which was clearly visible from SEM photography. But there some SiC fibers, which are formed on the surface of the graphite are not free-standing fibers rather than firmly stuck on the graphite surface (ribbon type morphology). So, there is an indication of two different types of mechanisms of fiber growth during the experiment.

The first mechanism is a gas phase reaction between SiO & CO for which the, inter laminar spacing of graphite flakes provides the reaction site given as:

$$SiO_{(g)} + 2CO_{(g)} \rightarrow SiC + CO_{2(g)} \text{.....(4.1)}$$

The second mechanism is a gas-solid reaction, probably taking place on the plane surface of graphite flakes, where the vapor pressure of CO is low due to very high radius of curvature. This type of morphology is not a free-standing fiber like -

structure rather than a ribbon or worm like structure, which is apparently attached to the plane surface of graphite flakes. This gas-solid reaction is given as

$$2SiO_{(g)} + 3C_{(s)} \rightarrow 2SiC + CO_{2\,(g)} \quad \text{.....(4.2)}$$

The weight percentage of silica and graphite taken and the temperature plays the vital role of forming the percentage of SiC. Graphite flake size and temperature are the two major controlling parameters to produce different kind of morphology of SiC.

The XRD results of reacted powder are presented below.

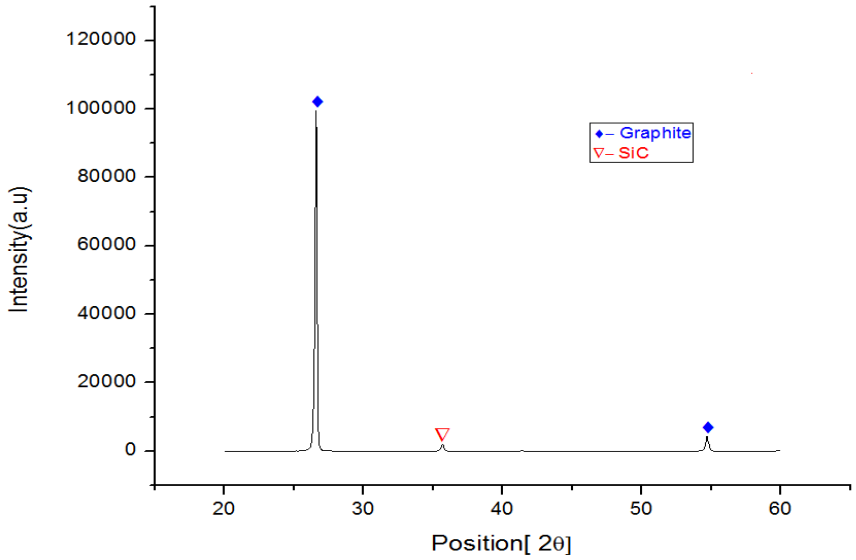
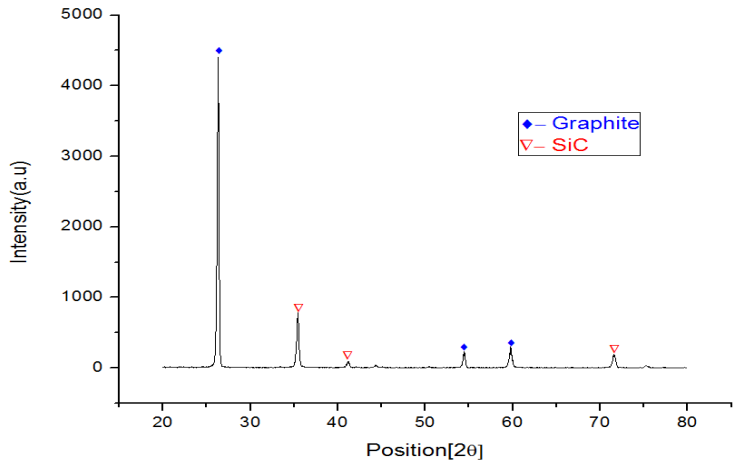
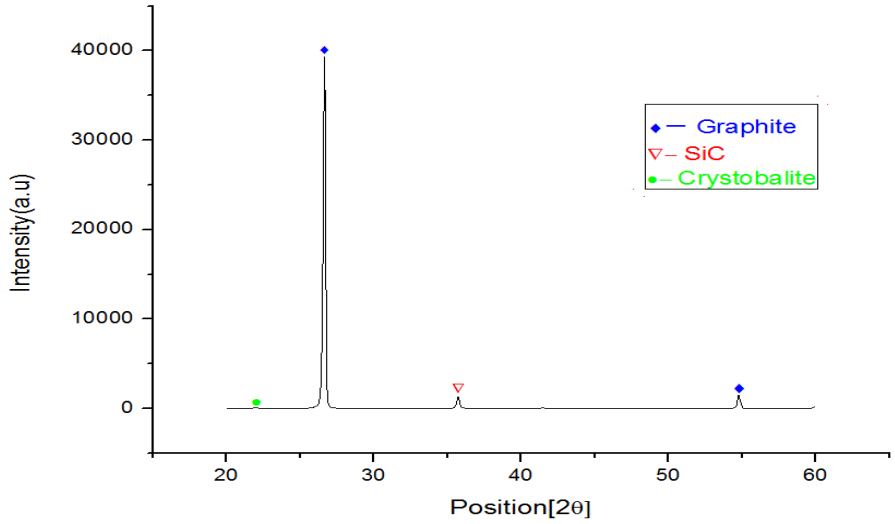



**Fig 4.1** XRD pattern of the sample prepared with 30 wt % microfine $SiO_2$ after control atmosphere thermal treatment @ 1640°C for 4h.




Graphite – 82.15 %, Silicon Carbide – 17.84 %, Crystobalite - 0 % (Intensity)

**Fig 4.2** XRD pattern of the sample prepared with 40 wt % microfine $SiO_2$ after control atmosphere thermal treatment @ 1640°C for 4h.




Graphite – 96.37 %, Silicon Carbide – 3.267 %, Crystobalite – 0.00035% (Intensity)

**Fig 4.3** XRD pattern of the sample prepared with 50 wt % microfine $SiO_2$ after control atmosphere thermal treatment @ 1640°C for 4h.

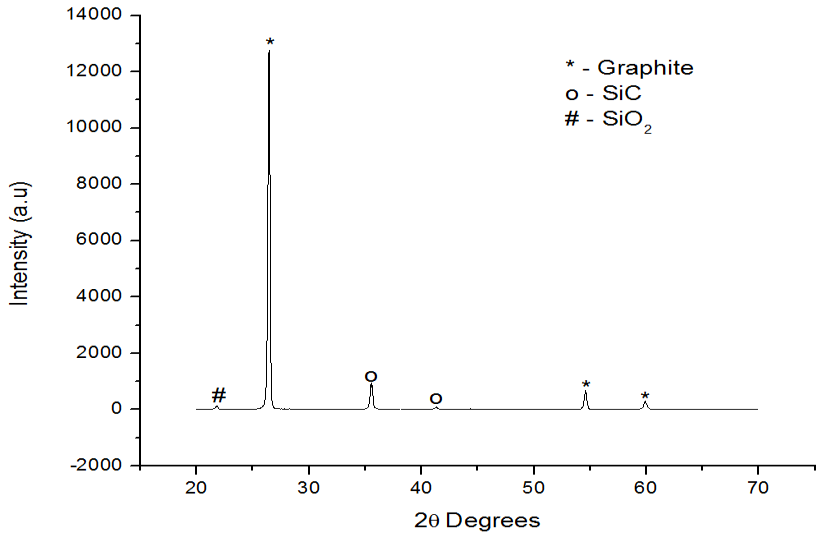



**Fig 4.4** XRD pattern of the sample prepared with 40 wt % microfine $SiO_2$ after control atmosphere thermal treatment @ 1580°C for 4h.

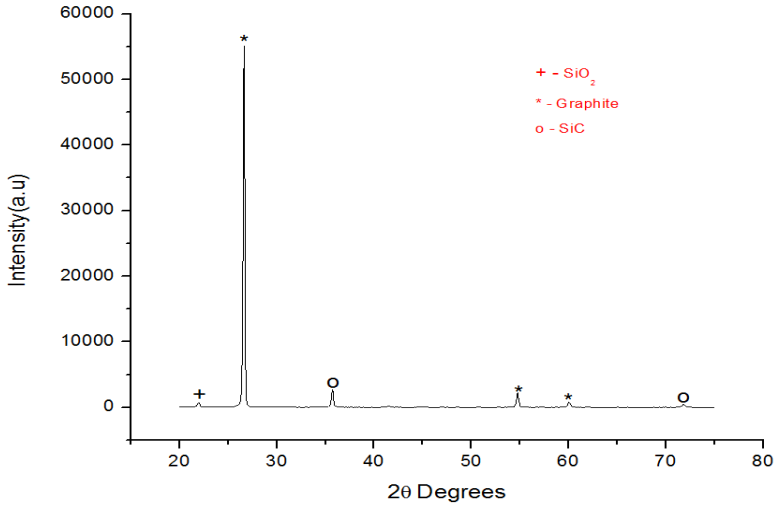



**Fig 4.5** XRD pattern of the sample prepared with 50 wt % microfine $SiO_2$ after control atmosphere thermal treatment @ 1580°C for 4h.

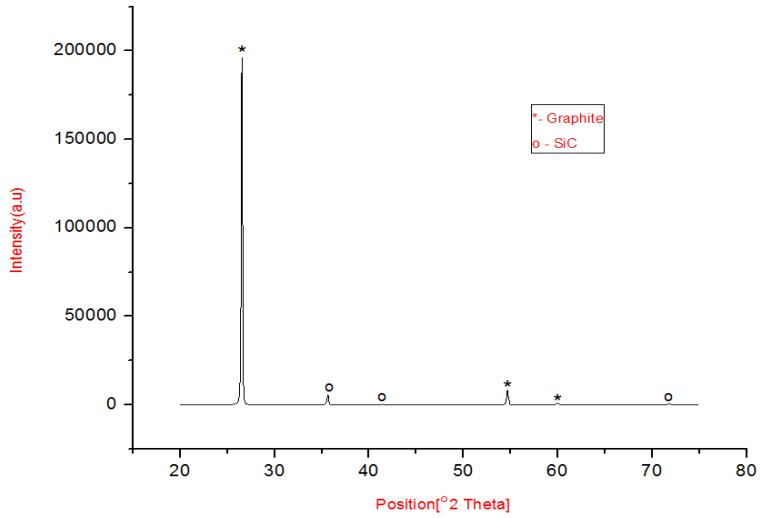



**Fig 4.6** XRD pattern of the sample prepared with 60 wt % microfine $SiO_2$ after control atmosphere thermal treatment @ 1580°C for 4h.

## 4.2 Morphology of Silicon Carbide formed:

During the experiments, there are two different kinds of morphologies of SiC formed by the reaction between graphite flake and micro-fine silica. As mentioned above, the fiber like morphology is produced at the edges of graphite flakes, and the irregular ribbon type morphology is formed on the flat surface of the graphite flakes. A number of samples were prepared of varying the weight percentage of graphite and silica and the temperature. The different kind of morphology of reacted powder can be clearly identified from SEM photographs given below.

### 4.2.1 SEM photographs of SiC with Fiber morphology:

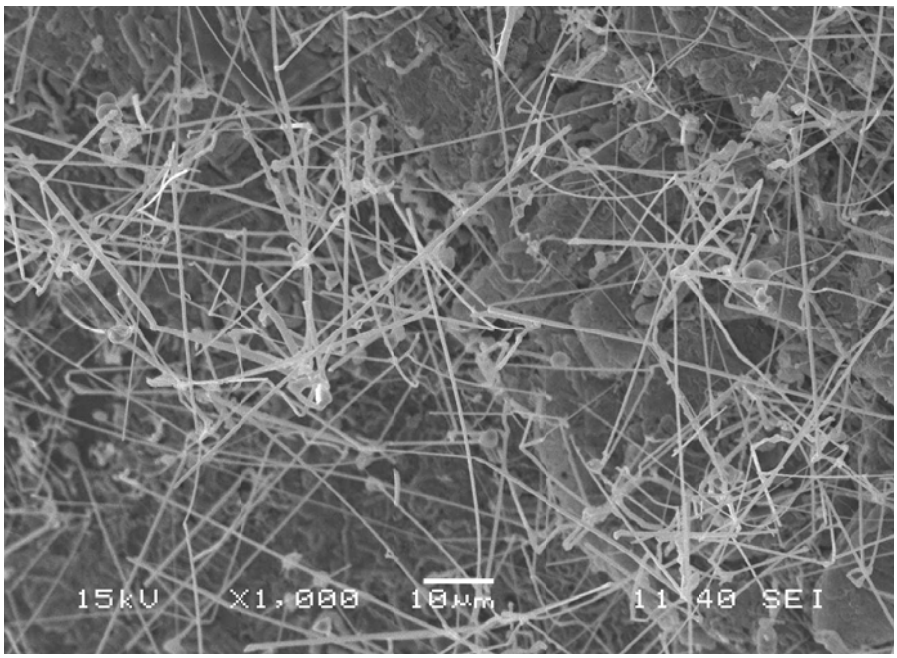



**Fig 4.7:** SEM pictures showing formation of both ribbons and fibers of β-SiC grown on graphite flakes.

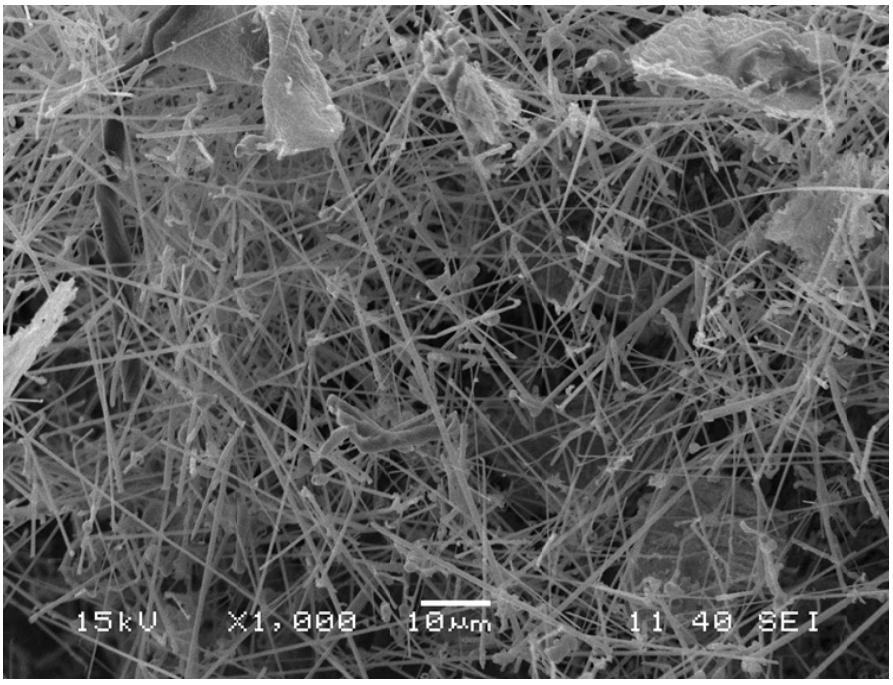



**Fig 4.8:** SEM pictures of only βSiC fiber grown on graphite flakes.

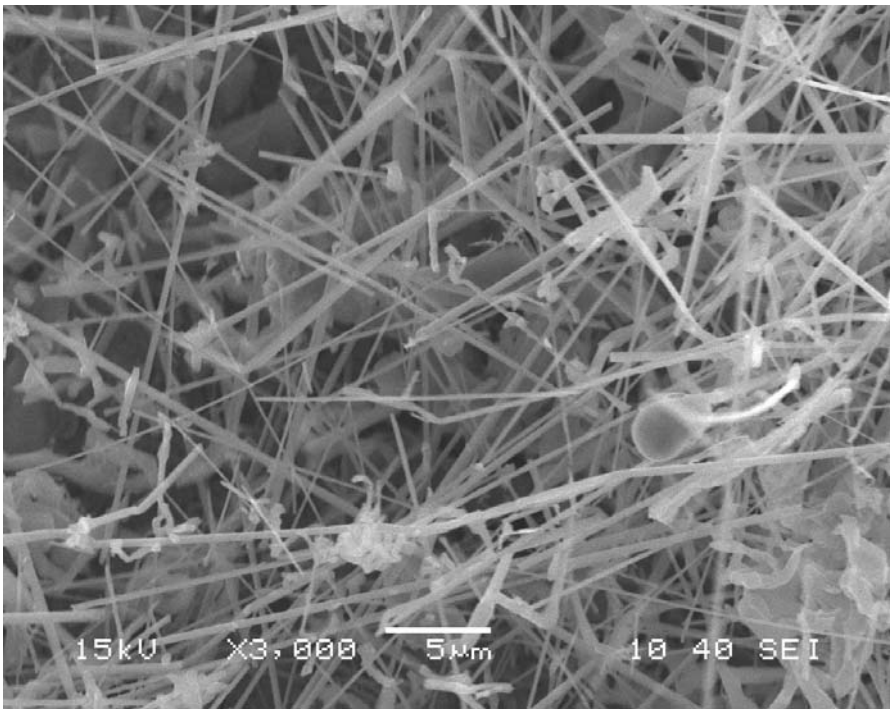



**Fig 4.9:** SEM photograph βSiC fiber grown on graphite flakes with varrying graphite to silica ratios.

## 4.2.2 SEM photographs of SiC with Ribbon morphology:

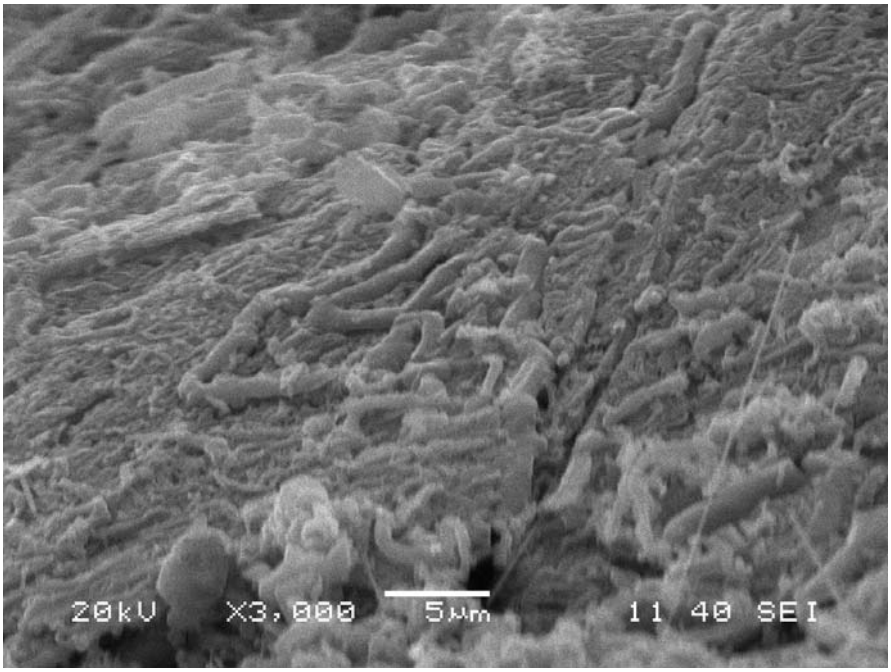



**Fig 4.10** SEM photograph βSiC ribbons grown on graphite flakes with 30% silica.

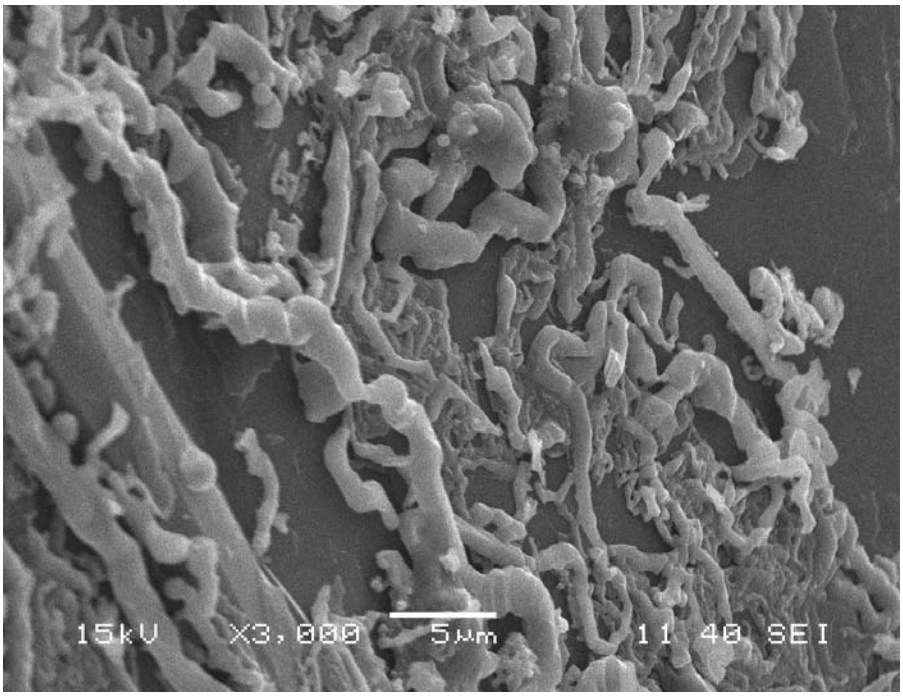



**Fig 4.11** SEM photograph βSiC ribbons grown on graphite flakes with 40% silica.

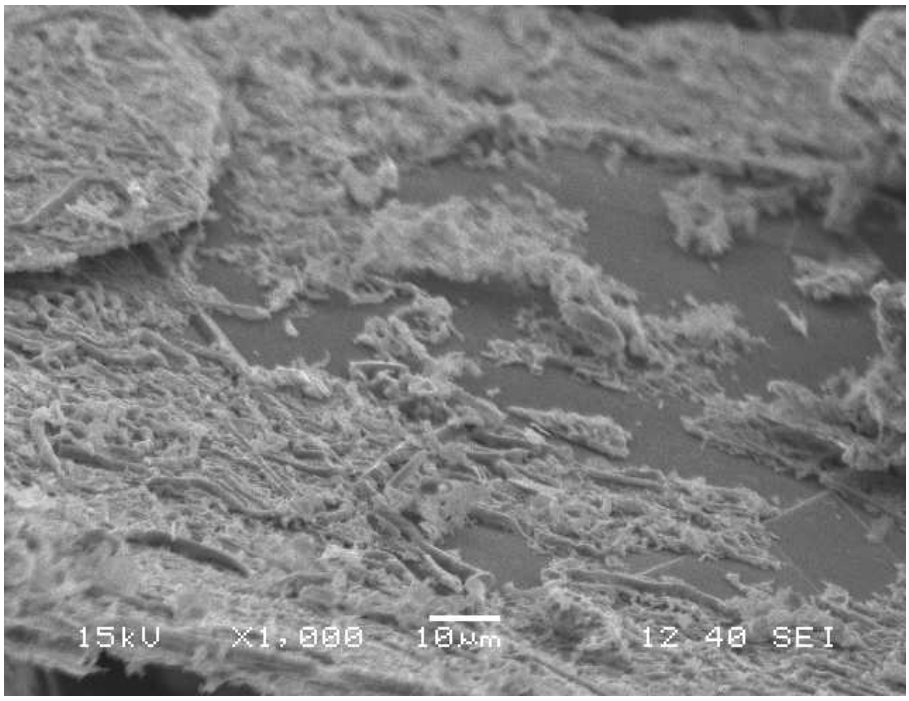



**Fig 4.12** SEM photograph βSiC ribbons grown on graphite flakes with 50% silica.

It has been observed that:

- When the rate of reaction is relatively low, then the fiber formation is predominant. The low reaction rate may be either due to relatively low firing temperature (< 1600°C), or due to the larger particle size of the graphite flakes.

- The affinity of fiber formation becomes more if the silica percentage is high (>40%). But if we go for higher percentage of silica (>55%), then there is more chance of presence of free silica in the composite powder, which has a detrimental property for refractory application.

- Generally, a good thick worm like ribbon structure is prone to form on the flat surface of the graphite flake, when the temperature is high (>1600°C). At low temperature, i.e (<1550°C), there is no formation of SiC from the flake graphite and micro-silica mixture.

## 4.3 CMOR analysis of MgO-C compacts with varying percentages of composites and other parameters:

The strength developed in the tempered MgO-C compacts mostly depends upon the sample preparation procedure and the weight percentage of materials used. So before analyzing the effects of composite on MgO-C compacts, we should focus on some of the parameters which influence the strength-property of the compacts.

**Table 4.1:** Binder percentage vs. Strength (CMOR)

| MATERIALS USED | BINDER PERCENTAGE | SAMPLES | DENSISTY (gm/cc) after tempering. | MOR ( Kg/cm²) | Avg. MOR (Kg/cm²) |
|---|---|---|---|---|---|
| MgO (-710µ) + BMF + 10% GRAPHITE + PITCH | 5% | 1a | 2.52 | 32.9 | 37.11 |
| | | 1b | 2.56 | 41.3 | |
| | 10% | 2a | 2.44 | 59.4 | 54.7 |
| | | 2b | 2.42 | 50.0 | |
| | 15% | 3a | 2.35 | 91.3 | 90.2 |
| | | 3b | 2.33 | 89.2 | |

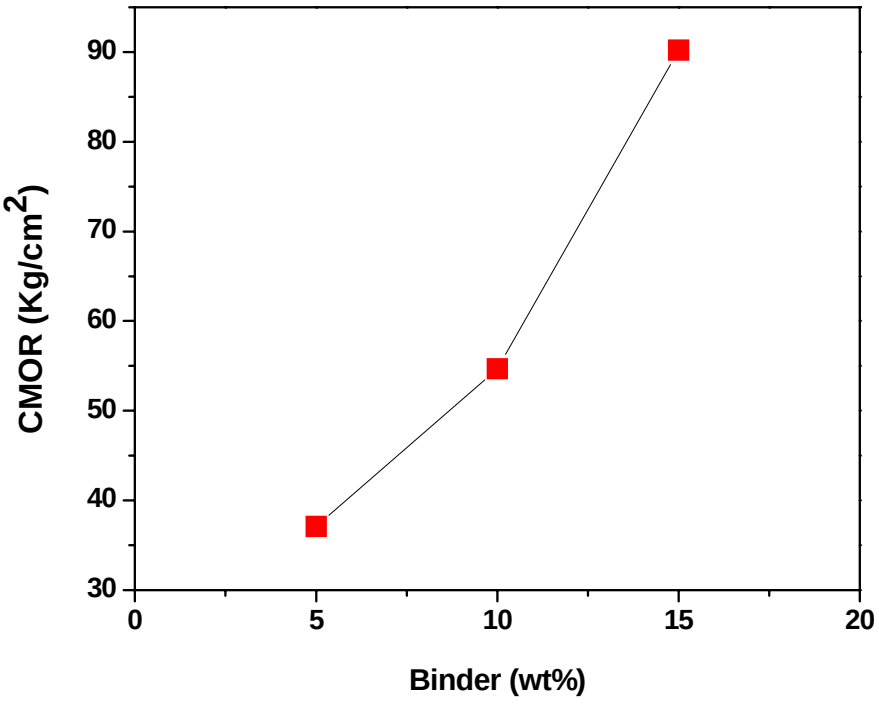



**Fig 4.13:** Variation of CMOR with binder content

From, the table 4.1 and fig 4.13, it is clearly known that, as the binder percentage is increased from 5% to 15%, the strength also increased. But during experimentation we came to know that as binder percentage exceeds 15% , the strength of the compacts are reduced after tampering, as bloating occurs in the tempered sample due to high amount of evolutions of organic gases from the binder.

**Table 4.2:** Time elapsed between hot mixing & pressing (in Minute)

| MATERIALS USED | SAMPLE NO. | Time elapsed between hot mixing & pressing (in Minute) | DENSITY (gm/cc) after tempering | MOR($Kg/cm^2$) |
|---|---|---|---|---|
| MgO (-710µ) (70%) + BMF(30%) + 10% GRAPHITE + 15% PITCH | 1 | AFTER 5 Min. | 2.32 | 62.735 |
| | 2 | AFTER 20 Min. | 2.32 | 54.078 |
| | 3 | Reheated & remixed (hot) And pressed after 5 min. | 2.33 | 113.4 |

From the above result, we can conclude that, the time elapsed between hot mixing and pressing is a crucial parameter for varying the strength of the MgO-C compacts. As the time, between hot mixing and pressing is increased, the binding property of solid pitch is decreased as the temperature decreases. Pitch has its binding property at higher temperature above 90°c. From the table, the MOR value of 3rd batch is higher which is due to aging of the hot mixture. During aging the polymerization of hot pitch takes place by developing the carbon-carbon bonds.

**Table 4.3:** Effects of adding **ribbon composite** to MgO-C compacts:

| MATERIALS USED | PERCENTAGE OF COMPOSITE | SAMPLES | DENSISTY ( gm/cc) after tempering | MOR ( $Kg/cm^2$) | Avg. MOR ($Kg/cm^2$) |
|---|---|---|---|---|---|
| MgO(-710µ) + BMF + GRAPHITE + **15% PITCH** | 5% | 1A | 2.30 | 67.41 | 60.155 |
| | | 1B | 2.28 | 52.9 | |
| | 10% | 2A | 2.27 | 33.87 | 32.35 |
| | | 2B | 2.25 | 30.83 | |
| | 15% | 3A | 2.26 | 69.7 | 71.6 |
| | | 3B | 2.27 | 73.53 | |

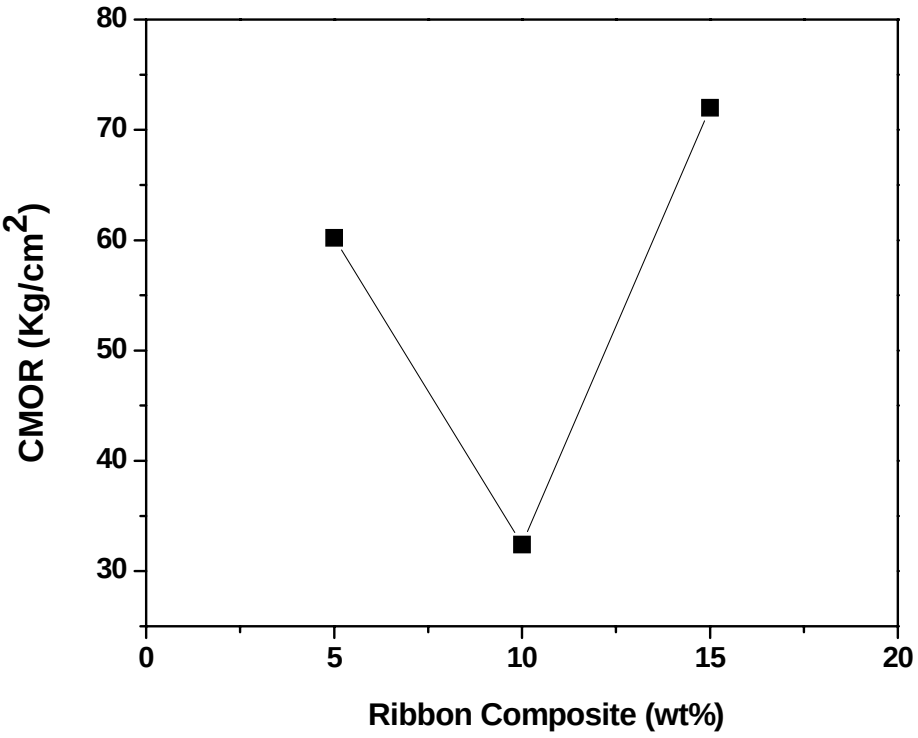



**Fig 4.14** Variation of CMOR with Percentage of ribbon composite

**Table 4.4:** Effects of adding Fiber **composite** to MgO-C compacts :

| MATERIALS USED | PERCENTAGE OF COMPOSITE | SAMPLES | DENSISTY (gm/cc) | MOR( Kg/cm$^2$) | Avg. MOR ( Kg/cm$^2$) |
|---|---|---|---|---|---|
| MgO (-710µ) + BMF + GRAPHITE + **15%** PITCH | 5% | 1a | 2.27 | 49 | 63.6 |
| | | 1b | 2.29 | 78.2 | |
| | 10% | 2a | 2.26 | 91.08 | 72.16 |
| | | 2b | 2.26 | 53.24 | |
| | 15% | 3a | 2.25 | 84.47 | 92.58 |
| | | 3b | 2.26 | 100.7 | |

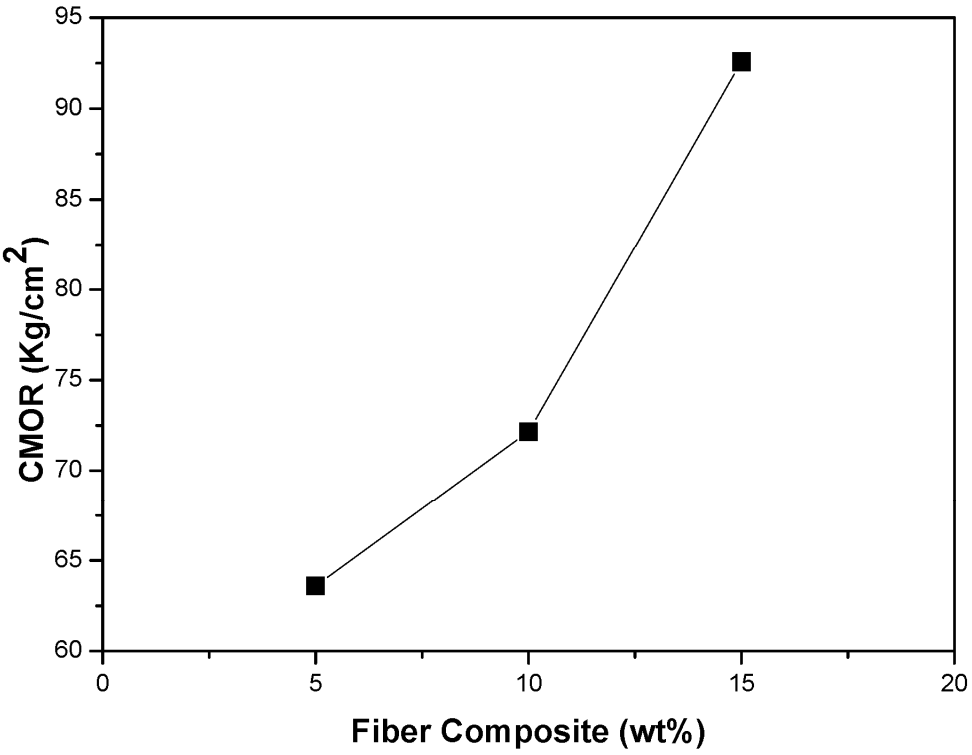



**Fig 4.15** Variation of CMOR with Percentage of fiber composite

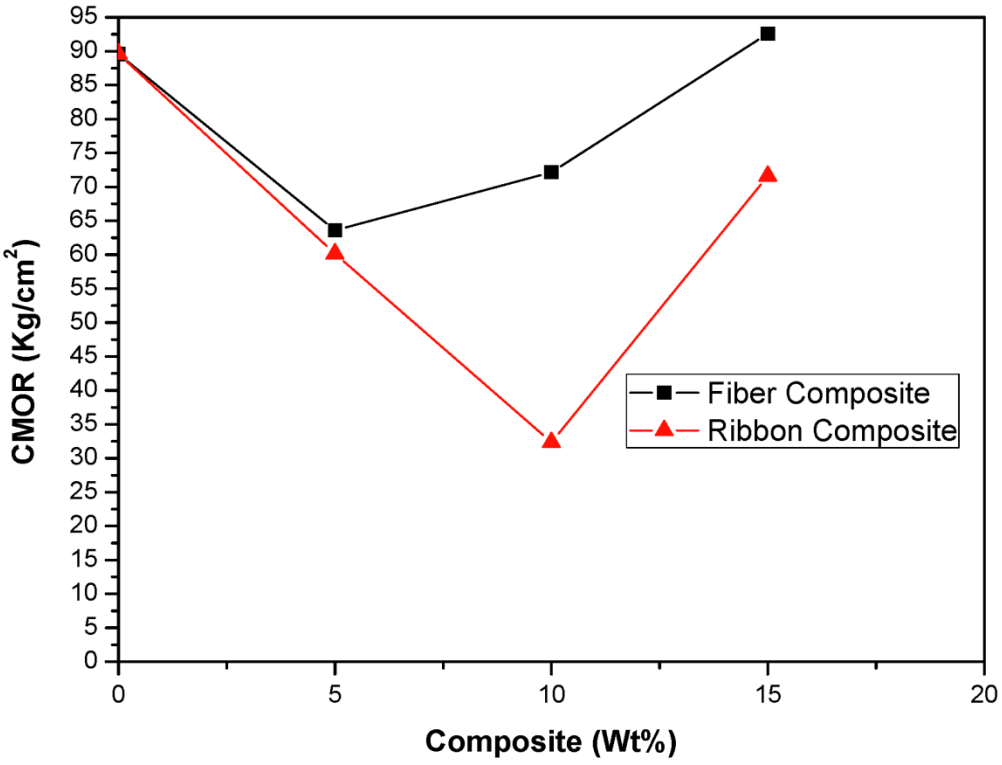



**Fig 4.16** Comparison the CMOR value between Ribbon & Fiber morphology

From the experimentation and the results presented in this project, it may be concluded that the strength giving properties of composites depend primarily upon two parameters i.e.

- Flow-ability of the binder (softened pitch) and also graphite-SiC micro composites during hot mixing followed by uniaxial pressing.

- Inherent strength of the graphite-SiC micro-composite particles.

From the analysis of different data presented in this project work, it may be concluded that addition of the composite particulates in small quantities significantly decreases the flow characteristics of the graphite flakes, which causes the decrease in strength of the compacts. Flow-ability problem is more significant in case of low binder content. However, with increasing binder content the flow characteristics improves and therefore the strength is increased up to about 15% binder (Fig. 4.13). Still higher binder content leads to swelling of the samples (during tempering) resulting in lowering of density as well as strength (data not presented).

It is evident from Fig. 4.16 that the strength of the compacts initially decreases with addition of the micro-composite, reaches a minimum but improves significantly with further addition of the composite powder. It is expected because the micro-composite with either β-SiC ribbon or the fiber is likely to have much greater inherent strength than pure graphite. So with increasing fraction of the micro-composite the problem of flow-ability is more than compensated by the inherently high strength of the micro-composite.

It is also interesting to note (Fig. 4.16) that the samples with SiC fiber possess higher strength than those with ribbons suggesting that the fibrous composite has a higher inherent strength than the composite with ribbon morphology. Optimization of the binder content and flow characteristics of the mix is, however important to observe this effect. In general, the flow characteristic of the fibrous composite is relatively poor and therefore one needs a careful sample preparation procedure to observe the beneficial effect of the inherent strength of the fibrous composite. Further work may be necessary in this direction.

# CHAPTER-5

# CONCLUSIONS & REFERENCES

## CONCLUSIONS:

In this project work, in particular, an attempt has been made to synthesize a graphite-SiC micro-composite by carbo-thermal reduction process and its effects on mechanical properties of magnesia-carbon refractory. The composite has been synthesized by in-situ formation of SiC on graphite through carbo-thermal reduction. There are two different morphologies of SiC formed by the reaction between graphite flake and micro-fine silica. As mentioned above, the fiber like morphology is produced at the edges of graphite flakes, and the irregular ribbon type morphology is formed on the flat surface of the graphite flakes. Our aim has been to improve the mechanical properties of MgO-C compacts by replacing a part of the graphite by this micro composite.

Batch processing sequence such as graphite coating on composite and pressing of hot mixture has significant influence on the ultimate flexural strength of developed composite material. A small variation particular pressing condition in processing reflects the tremendous change in the MOR value. Hence, the hot mixing process should be optimized in future. The strength of the MgO-C compacts depend upon two parameters, (a) Flow-ability of binder pitch and also the graphite-SiC micro composites during hot mixing, (b) Inherent strength of the composite particles. Therefore, the optimal binder percentage and the critical percentage of these graphite-SiC micro composite will enhance the strength more effectively. Further work will be necessary to optimize the preparation condition particularly to improve the flow-ability of the mix and also the percentage of the micro-composite to be used in the mix.

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